ge industrial facilities and sanitary

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Field Sampling of Residual Aviation Gasoline in Sandy Soil

by David W. Ostendorf, Lowell E. Leach, Erich S. Hinlein, and Yuefeng Xie

Abstract

Two complementary field sampling methods for the determination of residual aviation gasoline content in the contaminated capillary fringe of a fine, uniform, sandy soil were investigated. The first method featured field extrusion of core barrels into pint-size Mason jars, while the second consisted of laboratory partitioning of intact stainless steel core sleeves. The barrel extrusion procedure involved jar headspace sampling in a nitrogen-filled glove box, which delineated the 0.7m thick residually contaminated interval for subsequent core sleeve withdrawal from adjacent boreholes. Soil samples removed from the Mason jars (in the field) and sleeve segments (in the laboratory) were subjected to methylene chloride extraction and gas chromatographic analysis to compare their aviation gasoline content. The barrel extrusion sampling method yielded a vertical profile with 0.10m resolution over an essentially continuous 5.0m interval from the ground surface to the water table. The sleeve segment alternative yielded a more resolved 0.03m vertical profile over a shorter 0.8m interval through the capillary fringe. The two methods delivered precise estimates of the vertically integrated mass of aviation gasoline at a given horizontal location, and a consistent view of the vertical profile as well. In the latter regard, a 0.2m thick lens of maximum contamination was found in the center of the capillary fringe, where moisture filled all voids smaller than the mean pore size. The maximum peak was resolved by the core sleeve data, but was partially obscured by the barrel extrusion observations, so that replicate barrels or a half-pint Mason jar size should be considered for data supporting vertical transport analyses in the absence of sleeve partitions.

Introduction

Two complementary field sampling methods were studied for the determination of residual aviation gasoline content in a 22-year-old spill at the U.S. Coast Guard Air Station in Traverse City, Michigan. The observed distribution of light, separate phase hydrocarbons (LNAPLS) in the capillary fringe bears upon the larger problem of organic contamination of the subsurface environment.

The study site was underlain by a fine, uniform, sandy soil (mean grain size of 3.8×10^{-4}m in the capillary fringe), with a water table depth in excess of 5.0m. The field sampling program was conducted on June 21 and 22, 1990, at locations 50BS and 50BT as shown in Figure 1. The site presented an ideal case of residual non-aqueous phase liquid sampling for a number of reasons. The LNAPL resided above the shallow water table, and could be easily identified by elevated hydrocarbon vapor concentrations in the soil gas (Kampbell et al. 1990). Secondly, the Air Station was a U.S. Environmental Protection Agency research site, so that the hydrogeology (Twenter et al. 1985) and contamination (Ostendorf et al. 1989, Ostendorf 1990) were well documented. In the latter regard, the separate-phase aviation gasoline had migrated far downgradient of the original spill location and was confined to a thin (less than 1m thick) lens, distinguished by capillary ten-

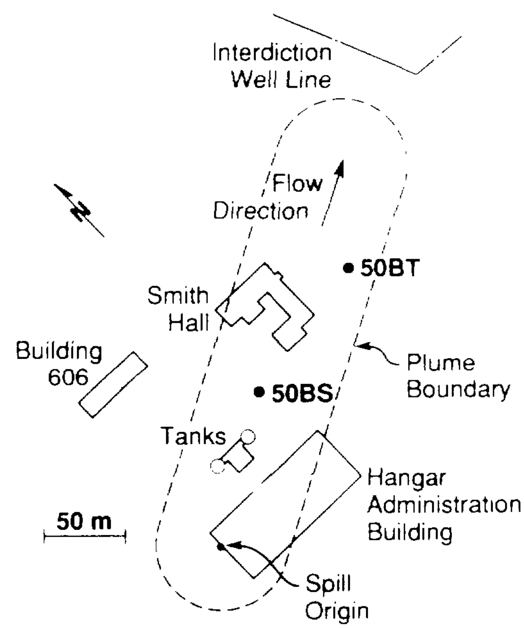


Figure 1. Site plan, U.S. Coast Guard Air Station, Traverse City, Michigan.

sion and subject to historical fluctuations of the water table. Thus, an appreciable fraction of the residual contamination could be recovered in a single core sleeve from a moderately shallow depth if the contaminated interval could be identified on an on-line basis. Finally, the residual product had been sampled and analyzed extensively, so that its composition, extraction protocol, and chromatography were also well established (Vandegrift and Kampbell 1988).

This paper describes a sampling technique designed to complement the existing U.S. Environmental Protection Agency and Institute of Ground Water Research core barrel extrusion protocol (Leach et al. 1988, Zapico et al. 1987) used at this and other sites in the past. Intact core sleeves were removed from adjacent (<2m distant) boreholes and partitioned in the laboratory for comparison with the field-extruded samples. The barrels yielded moderate vertical resolution (0.1m) but extended over the entire depth of the unsaturated zone and could be used in conjunction with an on-line headspace sampler to effectively delineate heavier zones of separate-phase contamination. The intact core sleeves delivered a finer (0.03m) vertical focus, but over a more limited (0.8m) interval. The chosen sleeve-sampled interval (as defined by the headspace results) corresponded to the contaminated capillary fringe at the site

Core Barrel Extrusion

The existing separate-phase studies at the site employed conventional U.S. Environmental Protection Agency and Institute of Ground Water Research soil sampling methodology. In summary, 0.102m I.D. hollow-stem augers were drilled to a prescribed depth and a 1.5m long, 0.0891m I.D. steam cleaned, Central Mine Equipment Co. (CME) thin-walled core barrel was percussion or hydraulically driven through the hollow-stem auger center into the underlying undisturbed soil. The augers were equipped with a clam-shell cap covering the hollow stem to prevent its blockage by heaving soil. The core barrel included a wireline piston to maintain a vacuum above the 0.916m soil sample, along with a pressure relief ball valve in the drive head and a cutting shoe fitted with a core retainer basket as indicated in Figure 2. The barrel sampler was disassembled by removing the drive head and piston. The core was then hydraulically extruded into autoclaved, wide-mouth Mason jars inside a (dry grade) nitrogen-filled glove box equipped with an iris to reduce site air contamination along the barrel. A flat spatula was used to pack the jars; typically 0.089m of barrel sample was fed into pint-size jars to analyze soil in the immediate vicinity of the capillary fringe. Quart-size Mason jars were used for regions beyond the fringe, where vertical resolution was not as critical to the study; 0.204m of barrel material was loaded into the larger jars. The Mason jars were sealed with bands and autoclaved lids as they were filled, and Kimwipes were used to clean the spatula between jars.

This extrusion protocol was augmented in this study by incorporating on-line headspace sampling of the Mason jar samples. The glove box was equipped with

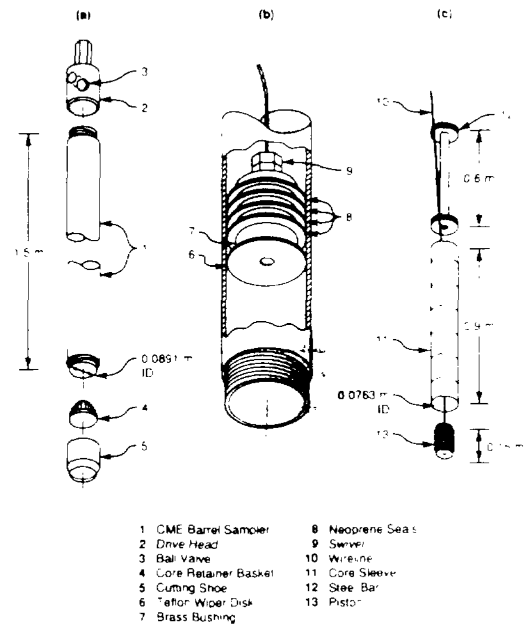


Figure 2. Field sampling devices: (a) CME thin-walled core barrel sampler; (b) wireline piston; (c) core sleeve.

copper gas lines welded to a Mason jar lid inside the box and attached to a portable gas meter outside, so that the headspace of the extruded sample could be analyzed. This procedure was run on-line with a Bacharach TLV combustible hydrocarbon meter, which was accurate to 10 ppm and had a range of 10 to 10,000 ppm on three scale settings. The sample jar was unscrewed inside the glove box, attached to the welded lid, and headspace sampled by the meter. The maximum vapor concentration was recorded to provide a rapid and qualitative measure of the degree of volatile hydrocarbon contamination in the soil sample. The observed meter readings varied from a background value of 10 ppm to off-scale levels (>10,000 ppm) associated with strong residual contamination in adjacent soil. Typically, the headspace analysis for the nine pint jars associated with a given core barrel could be run in 600s, allowing timely feedback to the rig crew on the placement of the next sampled interval.

Upon completion of the core barrel extrusion process, the quart and pint jars were subsampled in the glove box with a curved spatula rinsed with methylene chloride between uses. Roughly 0.025 and 0.01 kg of wet soil were placed into 2×10^{-5} m^3 volatile organic analysis (VOA) bottles equipped with screw caps and Teflon®-faced silicone closures for moisture and aviation gasoline determination, respectively. The gasoline VOA bottles were prelabeled and pretared with 5×10^{-6} m^3 deionized water to disaggregate the soil and 3×10^{-6} m^3 methylene chloride to dissolve the hydrocarbons. The field subsampling procedure was intended to stabilize the samples, thus minimizing evaporative losses and cross contamination during transport and storage. The VOA bottles were packed in

Observed	
I.D.	Depth Interv[illegible] Inches
Grain5	63.0-72.0
Grain6	117.0-126[illegible]
Grain11	138.0-146[illegible]
Grain9	154.0-162.0
Grain28	168.0-171.4
Grain26	174.5-178.0
Grain24	181.4-184.5
Avgas1	188.0-191.4
Avgas2	191.4-194.5
Avgas3,4	194.5-198.0
Avgas5	198.0-204.0
Avgas6,7	205.0-207.5
Avgas8	207.5-210.0
Avgas9	210.0-212.5
Avgas10	212.5-215.0
Avgas11	215.0-217.5
Avgas12,13	217.5-220.0
Avgas14	220.0-222.5
Avgas15	222.5-225.0
Avgas16	225.0-234.0
Avgas17	244.0-250.5
Avgas18	263.5-270.0

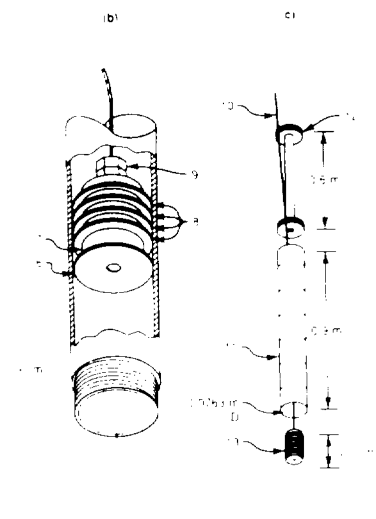
*) Precision in percent

ice until arrival at the University of M[illegible] mental Engineering Laboratory [illegible] chusetts, where they were stored a[illegible] proof refrigerator

Core Sleeve Partitioning

The 1.5m CME core barrel w[illegible] a 0.90m long, 0.0763m I.D., 0.00[illegible] steel intact core sleeve, prescored [illegible] it could be easily segmented by [illegible] adjustable pipe cutter. The CME [illegible] section and fitted with a coupling [illegible] the core sleeve after sample colle[illegible] tained a wireline piston and a [illegible] similar to the barrel components [illegible] 0.6m long steel bar used to mainta[illegible] in the barrel (Figure 2). The slee[illegible] long and featured four double nee[illegible] by brass bushings, and compress[illegible] wall of the sleeve with eight allen [illegible] of the piston. Once the compress[illegible] a Teflon wiper disk and stainle[illegible] screwed onto the end of the pist[illegible] sample from organics contained [illegible]

After the piston sleeve samp[illegible]



ME Barre Sampler / ve Head / il Valve / re Retainer Basket / ting Shoe / ston Wiper Disk / ass Bushing
8 Neoprene Seals / 9 Swivel / 10 Wireline / 11 Core Sleeve / 12 Steel Bar / 13 Piston

levices: (a) CME thin-walled core ve piston; (c) core sleeve.

welded to a Mason jar lid inside the d to a portable gas meter outside, so ace of the extruded sample could be rocedure was run on-line with a Bacha- ustible hydrocarbon meter, which was om and had a range of 10 to 10,000 ppm ettings. The sample jar was unscrewed box, attached to the welded lid, and ied by the meter. The maximum vapor is recorded to provide a rapid and quali- of the degree of volatile hydrocarbon n the soil sample. The observed meter from a background value of 10 ppm to (>10,000 ppm) associated with strong ination in adjacent soil. Typically, the sis for the nine pint jars associated with rel could be run in 600s, allowing timely rig crew on the placement of the next l.

the core barrel extrusion process, jars were subsampled in the glove box atula rinsed with methylene chloride ighly 0.025 and 0.01 kg of wet soil were 10^{-5} m^3 volatile organic analysis (VOA) with screw caps and Teflon®-faced sili- moisture and aviation gasoline deter- vely. The gasoline VOA bottles were tared with 5×10^{-6} m^3 deionized water he soil and 3×10^{-6} m^3 methylene chlo- the hydrocarbons. The field subsampling tended to stabilize the samples, thus mini- ve losses and cross contamination during orage. The VOA bottles were packed in

TABLE 1
Observed Moisture and Residual Aviation Gasoline Content

			Core Barrel 50BS Jars		
I.D.	**Depth Interval**	**Mid Interval**	**M**	**T**	**Headspace**
			kg moisture	**kg avgas**	
	Inches	**Depth z, m**	**kg wet soil**	**kg wet soil**	**ppm**
Grain5	63.0–72.0	1.72	0.031	<0.00001	60
Grain6	117.0–126.0	3.09	0.023	<0.00001	50
Grain11	138.0–146.0	3.61	0.047	<0.00001	50
Grain9	154.0–162.0	4.02	0.035	<0.00001	100
Grain28	168.0–171.4	4.31	0.038	<0.00002	720
Grain26	174.5–178.0	4.48	0.040	<0.00001	400
Grain24	181.4–184.5	4.66	0.039	<0.00001	550
Avgas1	188.0–191.4	4.83	0.033	0.00002	3000
Avgas2	191.4–194.5	4.91	0.034	0.00029	4200
Avgas3,4	194.5–198.0	4.99	0.032	0.00025(32)*	>10000
Avgas5	198.0–204.0	5.11	0.029	0.00032	4700
Avgas6,7	205.0–207.5	5.25	0.128	0.00022(41)	>10000
Avgas8	207.5–210.0	5.31	0.122	0.00031	3200
Avgas9	210.0–212.5	5.38	0.113	0.00043	3600
Avgas10	212.5–215.0	5.44	0.105	0.00049	3000
Avgas11	215.0–217.5	5.50	0.129	0.00130	>10000
Avgas12,13	217.5–220.0	5.57	0.122	0.00704(22)	>10000
Avgas14	220.0–222.5	5.63	0.133	0.00232	>10000
Avgas15	222.5–225.0	5.69	0.153	0.00098	2200
Avgas16	225.0–234.0	5.84	0.200	<0.00001	2500
Avgas17	244.0–250.5	6.29	0.126	<0.00001	220
Avgas18	263.5–270.0	6.79	0.143	<0.00001	80

*(Precision in percent.)

ice until arrival at the University of Massachusetts Environmental Engineering Laboratory in Amherst, Massachusetts, where they were stored at 4 C in an explosion-proof refrigerator.

Core Sleeve Partitioning

The 1.5m CME core barrel was modified to accept a 0.90m long, 0.0763m I.D., 0.00318m thick stainless steel intact core sleeve, prescored on 0.03m intervals so it could be easily segmented by a Rigid 0.05 to 0.1m adjustable pipe cutter. The CME barrel was cut at mid-section and fitted with a coupling for easy removal of the core sleeve after sample collection. The sleeve contained a wireline piston and a modified core basket similar to the barrel components, and also included a 0.6m long steel bar used to maintain its relative position in the barrel (Figure 2). The sleeve piston was 0.15m long and featured four double neoprene seals separated by brass bushings, and compressed against the inner wall of the sleeve with eight allen screws in the bottom of the piston. Once the compression was properly set, a Teflon wiper disk and stainless steel plate were screwed onto the end of the piston to protect the soil sample from organics contained in the seals.

After the piston sleeve sampler was assembled, it was steam cleaned, acetone washed, and air dried before deployment in the hollow-stem auger annulus. The sampler was lowered inside the auger column with center rods while maintaining minimum tension on the wireline to preserve the position of the piston. When the sleeve sampler contacted the clam-shell, the augers were lifted with the rods held fixed, thus opening the clam-shell and bringing the sleeve sampler to bear upon the upper surface of the undisturbed soil interval. The wireline was then pulled taut as the sleeve sampler was percussion or hydraulically driven into the soil, so that the piston was held stationary, creating a vacuum above the soil sample and preventing its escape as the device was lifted from the borehole. The core sleeves were stored upright in ice-packed vertical coolers until delivery to the explosion-proof refrigerator.

The core sleeves were partitioned in a vertical jig at the laboratory using the Rigid pipe cutter along their scores, which were rinsed with methylene chloride before separation. A methylene-chloride-rinsed, wide, flat spatula was slid through the completed cut, forming a sample base in the uppermost sleeve segment. The top end cap was removed and 0.025 and 0.010kg wet soil samples were withdrawn with a disposable syringe barrel and inserted into 2×10^{-5} m^3 VOA bottles for moisture and aviation gasoline content determination as was done for the extruded subsamples in the field. The partitioned sleeve segment was removed with the spatula after subsamples were taken and the end cap was replaced while the next lower segment was cut. The sleeve segment VOA bottles were stored at 4 C alongside their barrel extruded counterparts, and both sets of samples were extracted and analyzed identically from this point on, using the protocol described in the Appendix. Gravimetric and gas chromatographic analyses resulted in moisture and residual aviation mass contents [(M) and (T) respectively], as defined by:

$$M = \frac{\text{mass moisture}}{\text{wet soil mass}} \tag{1a}$$

$$T = \frac{\text{mass aviation gasoline}}{\text{wet soil mass}} \tag{1b}$$

Headspace Sampling and Comparative Method Precision

A single barrel extrusion and two adjacent (<2m distant) core sleeve boreholes were used to characterize location 50BS and 50BT (Figure 1), so that a total of six boreholes were drilled at the site. Headspace readings over the Mason jar samples were used to identify the contaminated soil region, as summarized by Table 1, Table 2, and Figure 3. The open circles in the figure denote the meter readings, which are seen to have registered high (including some off-scale) values over a fairly well-defined interval roughly 5.0m below the ground surface. The meter readings approached their maximum levels gradually with increasing depth through the unsaturated zone, reflecting a vertical gradient and an upward diffusive transport of hydrocarbon vapor concentration from the upper regions of the capillary fringe. The floor of separate phase contamination in the saturated soil was much more sharply defined by the headspace readings of Figure 3, due presumably to the buoyancy of LNAPL in ground water.

The open triangles in the figure represent the corresponding residual aviation gasoline content in the extruded Mason jar samples. A positive correlation existed between the headspace concentration and the residual aviation gasoline content, thus confirming the utility of the Bacharach hydrocarbon meter as an online indicator of vertical contamination. Indeed, the headspace readings were used in the field (without the benefit of the subsequently determined Mason jar extrusion data) to specify the intact core sleeve sampling intervals. The raw data at the two locations for the latter

TABLE 2
Observed Moisture and Residual Aviation Gasoline Content

Core Barrel 50BT Jars					
I.D.	Depth Interval Inches	Mid Interval Depth z, m	M kg moisture / kg wet soil	T kg avgas / kg wet soil	Headspace ppm
Grain9	57.0–66.0	1.56	0.041	<0.00001	10
Grain7	93.0–102.0	2.48	0.036	<0.00001	10
Grain11	129.0–138.0	3.40	0.040	<0.00001	60
Grain15	153.0–162.0	4.01	0.044	<0.00001	200
Grain23	162.0–165.0	4.16	0.086	<0.00001	750
Grain22	165.0–168.0	4.24	0.050	<0.00001	750
Avgas1	168.0–171.0	4.31	0.051	0.00029	4100
Avgas2	171.0–174.0	4.39	0.038	0.00058	5200
Avgas3	180.0–183.7	4.63	0.070	<0.00001	1800
Avgas4,18	183.7–187.5	4.73	0.053	0.00074(16)*	5400
Avgas5,6	187.5–191.2	4.82	0.075	0.00058(24)	3500
Avgas7	191.2–195.0	4.91	0.140	0.00191	2600
Avgas8,17	195.0–198.7	5.01	0.121	0.00137(7)	1800
Avgas9,10	198.7–202.5	5.10	0.136	0.00119(34)	>10000
Avgas11,12	202.5–206.2	5.20	0.136	0.00883(25)	>10000
Avgas13	206.2–210.0	5.30	0.190	0.00015	2000
Avgas14	216.0–223.0	5.59	0.104	<0.00001	240
Avgas16	227.4–229.6	5.81	0.129	<0.00001	90
Grain32	231.8–234.0	5.93	0.163	Not done	80
Grain10	234.0–240.0	6.03	0.180	Not done	20
Grain8	240.0–246.0	6.18	0.188	Not done	10

*(Precision in percent)

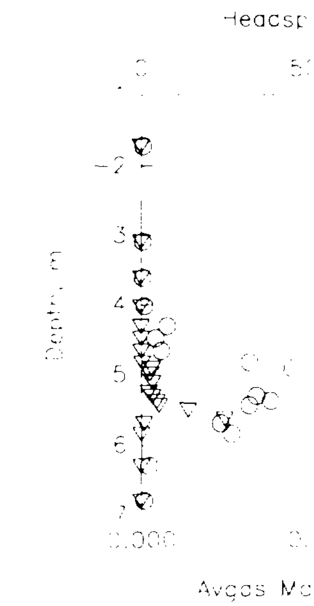


Figure 3. Barrel extruded residual avi... (b) 50BT. Triangles and circles repres...

Observed

I.D.	Depth Inte... Inches
7	200.0–20...
8, 9	201.2–20...
10	202.4–20...
11, 12	203.5–20...
13	204.7–20...
14, 15	205.9–20...
1, 16	207.1–20...
17, 18	208.3–20...
2, 19	209.5–2...
20, 21	210.6–2...
3, 22	211.8–21...
23, 24	213.0–21...
4, 25	214.2–2...
26, 27	215.4–2...
5, 28	216.5–21...
29, 30	217.7–21...
6, 31	218.9–22...
32, 33	220.1–22...
34	221.3–22...
35, 36	222.4–22...

*(Precision in percent

jar samples were used to identify region, as summarized by Table 1. 3. The open circles in the figure adings, which are seen to have ding some off-scale) values over a nterval roughly 5.0m below the meter readings approached their adually with increasing depth ed zone, reflecting a vertical gradi- iffusive transport of hydrocarbon rom the upper regions of the capil- r of separate phase contamination was much more sharply defined by igs of Figure 3, due presumably to APL in the ground water.

in the figure represent the corre- aviation gasoline content in the samples. A positive correlation headspace concentration and the oline content, thus confirming the ach hydrocarbon meter as an on- rtical contamination. Indeed, the were used in the field (without the uently determined Mason jar extru- the intact core sleeve sampling to the two locations for the latter

ne Content

T kg avgas / kg wet soil	Headspace ppm
<0.00001	10
<0.00001	10
<0.00001	60
<0.00001	200
<0.00001	750
<0.00001	750
0.00029	4100
0.00058	5200
<0.00001	1800
0.00074(16)*	5400
0058(24)	3500
0191	2600
0.00137(7)	1800
0.00119(34)	>10000
0.00883(25)	>10000
0.00015	2000
<0.00001	240
<0.00001	90
Not done	80
Not done	20
Not done	10

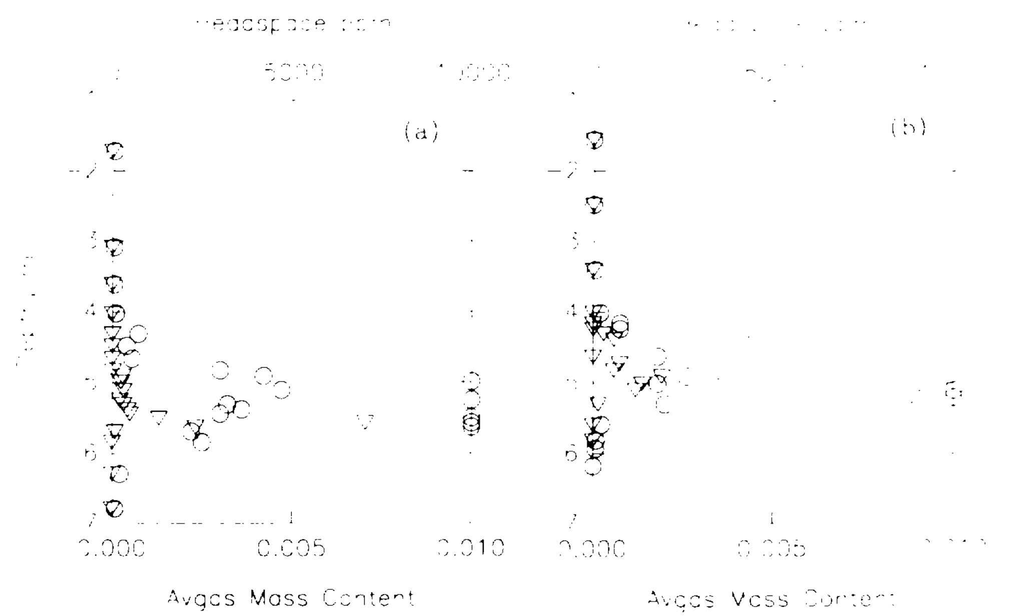


Figure 3. Barrel extruded residual aviation gasoline data and combustible hydrocarbon meter readings for locations (a) 50BS; and (b) 50BT. Triangles and circles represent residual aviation gasoline mass content (T) and jar headspace concentrations, respectively.

TABLE 3
Observed Moisture and Residual Aviation Gasoline Content

I.D.	Depth Interval Inches	Mid Interval Depth z, m	M kg moisture / kg wet soil	T kg avgas / kg wet soil
	Core Sleeve 50BS Profile 1			
7	200.0–201.2	5.10	0.033	<0.00001
8, 9	201.2–202.4	5.13	0.040	<0.00001
10	202.4–203.5	5.16	0.032	0.00002
11, 12	203.5–204.7	5.19	0.029	0.00021(43)*
13	204.7–205.9	5.22	0.035	0.00052
14, 15	205.9–207.1	5.25	0.032	0.00034(18)
1, 16	207.1–208.3	5.28	0.036	0.00049(32)
17, 18	208.3–209.5	5.31	0.032	0.00046(11)
2, 19	209.5–210.6	5.34	0.038	0.00033(32)
20, 21	210.6–211.8	5.37	0.036	0.00064(34)
3, 22	211.8–213.0	5.40	0.055	0.00046(7)
23, 24	213.0–214.2	5.43	0.068	0.00087(11)
4, 25	214.2–215.4	5.46	0.103	0.00062(8)
26, 27	215.4–216.5	5.49	0.117	0.00047(8)
5, 28	216.5–217.7	5.52	0.130	0.00085(9)
29, 30	217.7–218.9	5.55	0.132	0.00159(29)
6, 31	218.9–220.1	5.58	0.116	0.00084(5)
32, 33	220.1–221.3	5.61	0.119	0.00142(0)
34	221.3–222.4	5.64	0.118	0.00091
35, 36	222.4–225.0	5.69	0.136	0.00354(19)

*(Precision in percent.)

method are summarized in Tables 3–6.

Aviation gasoline sample replicate precision, defined by:

$$\text{precision} = \frac{\text{replicate value-replicate mean}}{\text{replicate mean}} \tag{2}$$

is cited for the sleeve and barrel data in Tables 1–6. The precision characterized the product uniformity in soil samples taken from the same segment or jar and so reflected sample variation across lateral scales of about 0.08 m and respective vertical scales of 0.03 m and 0.1 m. Table 7 summarizes the resulting statistics for the two sampling locations. The sleeve partitions were more precise than the barrels, with segment average precision varying from 13 to 23 percent, while the barrel range was 21 to 32 percent. The precision of the sleeve segment replicates suggested a relatively uniform horizontal distribution of the separate-phase gasoline, in keeping with the constancy of the capillary tension and resulting moisture content at a given elevation. By the same token, a strong vertical variation of tension and gasoline content was expected, so that the coarser vertical sampling interval of the jars gave rise to a decreased precision for the sampling method. This expectation was borne out by the statistics of Table 7.

Estimates of the mass (m) of aviation gasoline per horizontal area may be simply compared by summation of Tables 1–6 over the depth increments (H)

$$m = \frac{\text{mass aviation gasoline}}{\text{horizontal area}} \tag{3a}$$

$$m = \Sigma(\rho_B T H) \tag{3b}$$

with wet soil bulk density (ρ_B) computed in accordance with:

$$\rho_B = \frac{\text{wet soil mass}}{\text{total volume}} \qquad (n = 0.367) \tag{4a}$$

$$\rho_B = \rho_S \left(\frac{1 - n}{1 - M} \right) \qquad (\rho_S = 2650 \text{ kg/m}^3) \tag{4b}$$

Ostendorf (1990) estimated the porosity (n) based on earlier coring work at the site, and the solid grain density (ρ_S) was appropriate for quartz sands. The sleeve segments and Mason jars yielded respective mass estimates of 1.40 and 1.77 kg/m^2 at location 50BS and 3.71 and 2.82 kg/m^2 at 50BT. The replicate precision of 12 percent and 14 percent for these estimates was excellent, and suggested that the continuous pint-size Mason jar profile resolution of 0.1m was sufficient to accurately determine

TABLE 4
Observed Moisture and Residual Aviation Gasoline Content

Core Sleeve 50BS Profile 2

I.D.	Depth Interval Inches	Mid Interval Depth z, m	M kg moisture / kg wet soil	T kg avgas / kg wet soil
7	185.0–186.2	4.72	0.027	<0.00001
8, 9	186.2–187.4	4.75	0.032	<0.00001
10	187.4–188.6	4.78	0.036	<0.00002
11, 12	188.6–189.7	4.81	0.058	0.00001
13	189.7–190.9	4.84	0.141	0.00139
14, 15	190.9–192.1	4.87	0.074	0.00016(31)*
16	192.1–193.3	4.90	0.050	0.00002
17, 18	193.3–194.5	4.93	0.069	0.00028(30)
1, 19	194.5–195.6	4.96	0.039	0.00011(36)
20, 21	195.6–196.8	4.99	0.031	0.00050(18)
2, 22	196.8–198.0	5.02	0.042	0.00059(27)
23, 24	198.0–199.2	5.05	0.039	0.00023(12)
3, 25	199.2–200.4	5.08	0.038	0.00005
26, 27	200.4–201.5	5.11	0.044	0.00024(8)
4, 28	201.5–202.7	5.14	0.040	0.00035(3)
29, 30	202.7–203.9	5.17	0.048	0.00065(23)
5, 31	203.9–205.1	5.20	0.057	0.00071(7)
32, 33	205.1–206.3	5.23	0.062	0.00104(2)
6, 34	206.3–207.4	5.26	0.072	0.00221(24)
35, 36	207.4–210.0	5.31	0.077	0.00112(12)

*(Precision in percent)

TABLE 5
Observed Moisture and Residual Aviation Gasoline Content

Core Sleeve 50BT Profile 1				
I.D.	**Depth Interval** **Inches**	**Mid Interval** **Depth z, m**	**M** **kg moisture / kg wet soil**	**T** **kg avgas / kg wet soil**
4	190.2–191.4	4.85	0.056	0.00009
5	191.4–192.6	4.88	0.053	0.00435
6	192.6–193.7	4.91	0.049	0.00158
7	193.7–194.9	4.94	0.049	0.00154
8, 9	194.9–196.1	4.97	0.123	0.00416(2)*
10	196.1–197.3	5.00	0.095	0.00296
11, 12	197.3–198.5	5.03	0.077	0.00330(6)
13	198.5–199.6	5.06	0.067	0.00242
14, 15	199.6–200.8	5.09	0.078	0.00820(8)
16	200.8–202.0	5.12	0.109	Not Done
17, 18	202.0–203.2	5.15	0.092	0.00188(32)
19	203.2–204.4	5.18	0.096	0.00374
20, 21	204.4–205.5	5.21	0.136	0.00596(33)
22	205.5–206.7	5.24	0.155	0.00002
23, 24	206.7–207.9	5.27	0.164	0.00001
25	207.9–209.1	5.30	0.161	<0.00001
26, 27	209.1–210.3	5.33	0.155	<0.00001
[illegible], 28	210.3–211.4	5.36	0.142	0.00203(24)
29, 30	211.4–212.6	5.39	0.148	0.00093(54)
2, 31	212.6–213.8	5.42	0.144	<0.00001
32, 33	213.8–214.9	5.45	0.138	<0.00001

*(Precision in percent.)

the vertically integrated mass of residual aviation gasoline at a given location.

Vertical Profile Structure

The two field data bases were used in a complementary fashion to assess the vertical distribution of the LNAPL and moisture. In the latter regard, Tables 1-6 suggest that the aviation gasoline content was far smaller than the water content in the soil at Traverse City. The separate-phase product was consequently ignored in the analysis of the coherent vertical moisture profile evident in both the extruded and partitioned data. The (volumetric) degree of water saturation (S_W) then defined the capillary fringe:

$$S_W = \frac{\text{water volume}}{\text{void volume}} \tag{5a}$$

$$S_W = \frac{\rho_B\,(M-T)}{\rho_W n} \qquad (\rho_W = 1000\ \text{kg/m}^3) \tag{5b}$$

with water density (ρ_W).

The water saturation was accordingly construed as the cumulative density function of the pore size (r) (Mualem 1976), described by the empirical distribution of Van Genuchten (1980):

$$S_W = \left[1 - \left(\frac{r_M}{r}\right)^{\alpha}\right]^{\frac{1}{\alpha}-1} \qquad (\alpha > 1) \tag{6a}$$

$$r = \frac{2\sigma_T}{\rho_W g (z_{WT} - z)} \qquad (\sigma_T = 0.072\ \text{N/m}) \tag{6b}$$

with mean pore size (r_M), uniformity exponent (α), and depth (z) below the ground surface. The pore size, in turn, was simply related to the capillary tension, which was proportional to the vertical distance above the water table. The water table depth (z_{WT}), gravitational acceleration (g), and surface tension (σ_T) appear in Equation 6, with the latter parameter measured for contaminated site water in air by the drop-weight method (Harkins and Brown 1919) in the laboratory.

The uniformity parameter, water table depth, and mean pore size were calibrated at each location using a nested Fibonacci search (Beveridge and Schechter 1970) to minimize the mean ($\bar{\delta}$) and standard deviation (σ) of the error (δ) defined by Benjamin and Cornell

TABLE 6
Observed Moisture and Residual Aviation Gasoline Content

		Core Sleeve 50BT Profile 2		
I.D.	Depth Interval	Mid Interval	M	T
			kg moisture	kg avgas
	Inches	Depth z, m	kg wet soil	kg wet soil
5	178.6–179.8	4.56	0.051	0.00008
6	179.8–181.0	4.59	0.050	<0.00001
7	181.0–182.1	4.62	0.033	<0.00001
8, 9	182.1–183.3	4.65	0.034	<0.00001
10	183.3–184.5	4.68	0.036	<0.00001
12	184.5–185.7	4.71	0.050	0.00066
13	185.7–186.9	4.74	0.051	0.00100
14, 15	186.9–188.0	4.77	0.057	0.000161(24)*
16	188.0–189.2	4.80	0.051	0.00163
17, 18	189.2–190.4	4.83	0.110	0.00169(6)
19	190.4–191.6	4.86	0.103	0.00163
20, 21	191.6–192.8	4.89	0.113	0.00092(3)
22	192.8–194.0	4.92	0.096	0.00107
23, 24	194.0–195.1	4.95	0.104	0.00122(1)
1, 25	195.1–196.3	4.98	0.159	0.00115(25)
26, 27	196.3–197.5	5.01	0.120	0.00176(30)
2, 28	197.5–198.7	5.04	0.127	0.00225(6)
29, 30	198.7–199.9	5.07	0.138	0.00564(2)
31	199.9–201.0	5.10	0.123	0.00996(7)
32, 33	201.0–202.2	5.13	0.120	0.01465(19)
4, 34	202.0–203.4	5.16	0.127	0.01705(8)
35, 36	203.4–205.4	5.20	0.154	0.00535(25)

*(Precision in percent)

(1970) as

$$\delta = S_W \text{ (measured)} - S_W \text{ (predicted)} \quad (7a)$$

$$\bar{\delta} = \frac{1}{J}\Sigma(\delta) \quad \text{(J samples)} \quad (7b)$$

$$\sigma = \left[\frac{1}{J}\Sigma(\delta^2) - \bar{\delta}^2\right]^{1/2} \quad (7c)$$

The data below the 4m depth were used in the pore size optimization, since capillarity and not infiltration dominated the moisture regime near the water table. Table 8 and Figure 4 summarize the calibration results. The 15 percent standard deviation indicated modest accuracy, reflective of errors associated with field sampling of a periodically varying water table subject to infiltration. The close correspondence of the calibrated mean pore sizes and uniformities at the two locations was encouraging and attested to the homogeneity of the soil at Traverse City. In this regard, the α values compared favorably with Haverkamp and Parlange's (1986) grain size-based estimate of 2.36 (Ostendorf and Kamp-

TABLE 7
Residual Aviation Gasoline Sample Precision

I.D.	Type	Number of Replicates	Precision %
50BS Profile 1	Sleeve	15	18
50BS Profile 2	Sleeve	13	18
50BS Jars	Barrel	3	32
50BT Profile 1	Sleeve	7	23
50BT Profile 2	Sleeve	12	13
50BT Jars	Barrel	5	21

TABLE 8
Analysis of Moisture and Residual Aviation Gasoline Contents

Location	z_{WT} m	α	r_M m	z_M m	σ %
50BS	6.00	2.68	3.53 ± .07	5.55	18
50BT	5.60	2.23	3.47 ± [illegible]	5.23	1[illegible]

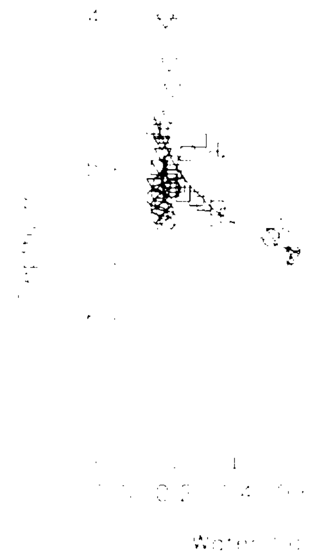


Figure 4. Fitted Van Genuchten (1980) p
(squares) water saturation (S_W) data at lo
sketched.

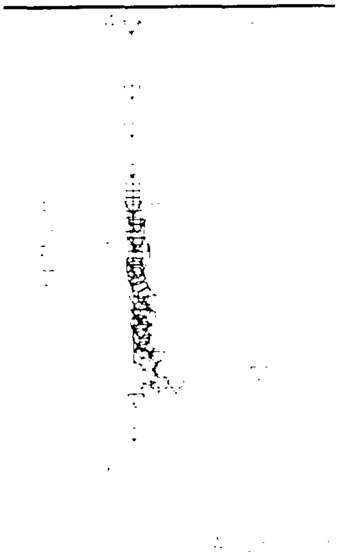


Figure 5. Observed aviation gasoline satu
(a) 50BS: and (b) 50BT. Overlapping slee
also sketched.

bell 1990)

Figure 5 displays the residual av
aration (S_G) defined on a volumetr

$$S_G = \frac{\text{volume gasoline}}{\text{void volume}}$$

$$S_G = \frac{\rho_B T}{\rho_G n} \quad (\rho_G = 707 \text{ kg}$$

with aviation gasoline density (ρ_G
1986). It was reassuring to note

line Content

M oisture et soil	T kg avgas / kg wet soil
051	0.00008
050	<0.00001
033	<0.00001
034	0.00001
036	<0.00001
050	0.00066
052	0.00100
057	0.00161(24)*
051	0.00163
110	0.00169(6)
103	0.00163
113	0.00092(3)
096	0.00107
104	0.00122(1)
	0.00115(25)
	0.00176(30)
127	0.00225(6)
138	0.00564(2)
123	0.00996(7)
120	0.01465(19)
127	0.01705(8)
154	0.00535(25)

TABLE 7
tion Gasoline Sample Precision

Type	Number of Replicates	Precision %
Sleeve	15	18
Sleeve	13	18
Barrel	3	32
e	7	23
eeve	12	13
Barrel	5	21

TABLE 8
isture and Residual Aviation asoline Contents

r	r_M m	z_M m	σ %
2.68	3.53×10^{-5}	5.58	15
2.23	3.97×10^{-5}	5.23	16

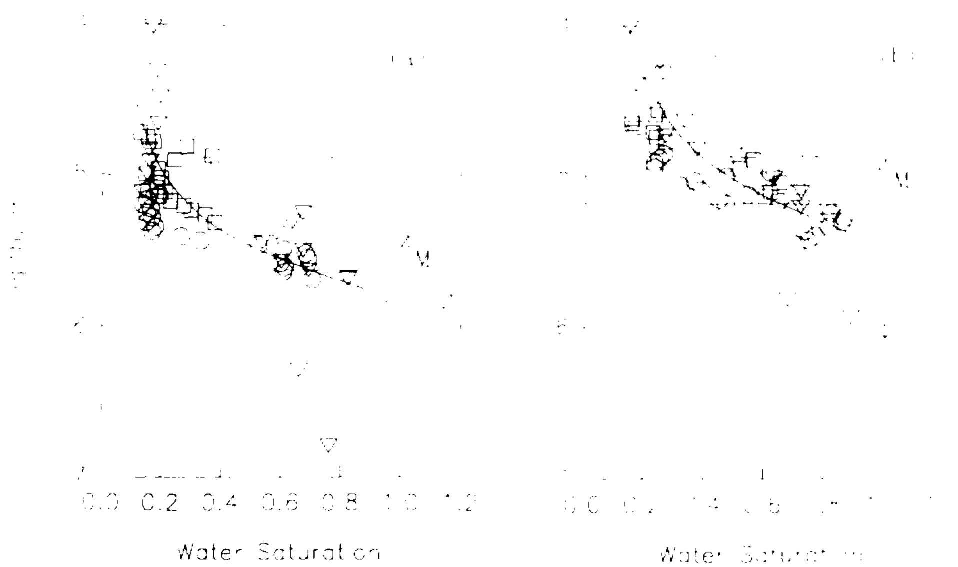


Figure 4. Fitted Van Genuchten (1980) pore size distribution (curves) through jar (triangles), profile 1 (circles), and profile 2 (squares) water saturation (S_W) data at locations (a) 50BS; and (b) 50BT. Elevation (z_M) of mean pore size saturation is also sketched.

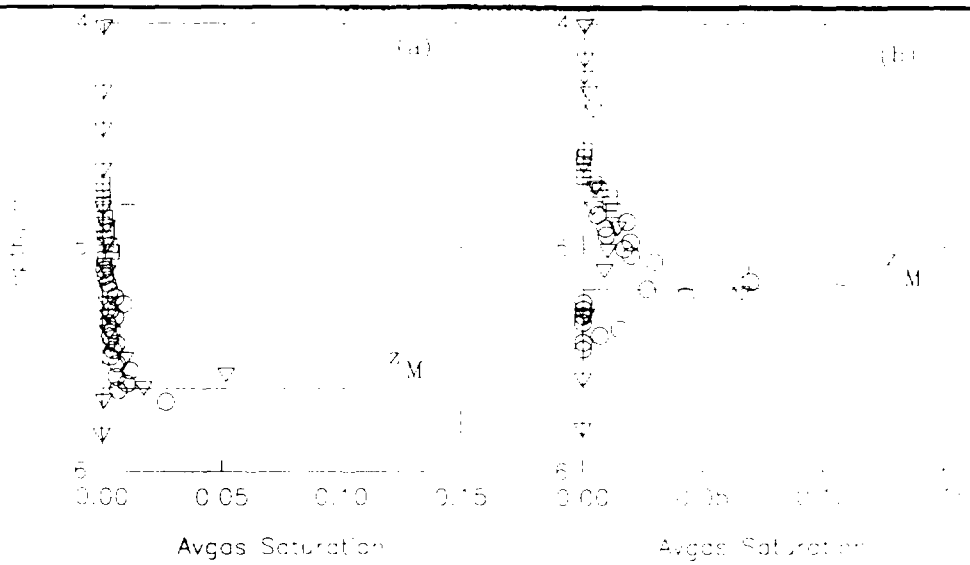


Figure 5. Observed aviation gasoline saturation (S_G) for jar (triangles), profile 1 (circles), and profile 2 (squares) data at locations (a) 50BS; and (b) 50BT. Overlapping sleeve data are averaged, and appear as circles. Elevation (z_M) of mean pore size saturation is also sketched.

bell 1990).

Figure 5 displays the residual aviation gasoline saturation (S_G) defined on a volumetric basis by:

$$S_G = \frac{\text{volume gasoline}}{\text{void volume}} \tag{8a}$$

$$S_G = \frac{\rho_B T}{\rho_G n} \qquad (\rho_G = 707 \text{ kg/m}^3) \tag{8b}$$

with aviation gasoline density (ρ_G) (Ostendorf et al. 1989). It was reassuring to note a consistent vertical distribution of product implied by barrel extrusion and sleeve partition methods. Taken together, the jars and segments defined an overall residually contaminated thickness of about 0.7m, with a 0.2m thick embedded lens of heavier contamination. The lens of maximum concentration occurred at the depth z_M through the middle of the capillary fringe, defined analytically by substituting r_M for r in Equation 6b with the result

$$z_M = z_{WT} - \frac{2\sigma_I}{\rho_W g r_M} \qquad (r = r_M) \tag{9}$$

and cited in Table 8. This elevation marked the saturation of all pores smaller than the mean pore size, and its status as the locus of peak pollution is displayed on Figure 5.

Discussion

The recommended field sampling protocol for residual LNAPL contamination depends on the intended use of the data. Core barrel extrusion into pint-size Mason jars was relatively rapid, and is the method of choice for vertically integrated data, with the headspace sampling protocol included for on-line specification of maximum contamination intervals. The efficient determination of mass values facilitates sampling at numerous horizontal locations for studies detailing the lateral extent of separate-phase pollution.

Vertically defined profiles for volatilization or dissolution model data bases should be obtained with the complementary use of both sampling methods. The intact core sleeves were labor-intensive in the field and laboratory, and should be used judiciously for a vertically resolved data base (0.03m intervals) at a few key locations. Figure 5 implies that the barrel extrusion data defined much of the vertical structure of the residual aviation gasoline distribution as well; although the maximum contamination thickness of 0.2m approached the method resolution of 0.1m at Traverse City. Thus the details of peak concentration were somewhat obscured by the pint-size jars, and the complementary use of the core sleeves is well advised. In the absence of sleeve partitions, half-pint Mason jar sizes should be considered in zones of peak concentration, perhaps with replicate barrel extruded boreholes for vertically defined data bases. In this regard, half-pint Mason jars retain about 0.05 m of the barrel and would accordingly increase the resolution and precision of barrel extruded data, where permitted by constraints of field sampling and laboratory analytical loads.

The observed LNAPL mass content values, which did not exceed 0.02kg aviation gasoline/kg wet soil, were well below residual gasoline values of 0.03 to 0.05 attained in laboratory prepared columns with fine, uniform sand (at field moisture capacity) and automobile gasoline (Hoag and Marley 1986). It was accordingly suspected that the fuel at Traverse City existed primarily as a discontinuous separate phase, although a definitive conclusion would require an undisturbed thin section detailing the microstructure of the product distribution. At the very least, the existence of field concentrations far below laboratory column residuals for soil types similar to that at Traverse City suggests that caution be exercised in the scale-up of transport coefficients and mass fluxes from artificially contaminated soils to field applications.

The coincidence of the independently determined mean pore size position with the maximum lens of LNAPL concentration suggests that the residual aviation gasoline distribution may be predicted as a function of moisture content on theoretical grounds. A modeling effort is currently underway to explore this correlation. The gas chromatograms generated in accordance with the LNAPL analysis provided concentrations of individual aviation gasoline constituents, as discussed in the Appendix. Future model studies of the vertical distribution of the separate-phase components are contemplated for the Traverse City data base as well, in an attempt to assess the impact of solubility and vapor pressure characteristics on the transport of LNAPL through the capillary fringe (Baehr 1987).

Summary and Conclusions

Two complementary field sampling methods for determining the residual aviation gasoline content in the contaminated capillary fringe of a fine, uniform, sandy soil were investigated. The first method featured field extrusion of cored material (collected with a barrel sampler) into pint-size Mason jars, augmented by on-line sampling of jar headspace vapors in a nitrogen-filled glove box. The combustible hydrocarbon meter successfully delineated the residually contaminated interval (0.7m thick at a depth in excess of 5m) for subsequent intact stainless steel core sleeve sampling, which comprised the second field method. The sleeves were partitioned under more controlled laboratory conditions.

Both barrel and sleeve soil subsamples were subjected to methylene chloride extraction and gas chromatography, an analysis yielding surprisingly precise estimates of the vertically integrated gasoline mass and the maximum contamination location in the capillary fringe. Integrated mass precisions of 12 percent and 14 percent were attained at the two sampling locations and, in the latter regard, the locus of peak separate-phase pollution coincided closely with the elevation where all pores smaller than the mean pore size were saturated. This lens of peak concentration was about 0.2m thick at both locations, so that the sleeve resolution of 0.03m was sufficient to resolve its vertical structure. The 0.1m barrel resolution may have obscured some details of the maximum profile, so that replicate boreholes or half-pint sizes may be warranted for vertical transport modeling in the absence of companion sleeve data. A single borehole core extrusion with pint-size jars was judged adequate for depth-integrated, horizontally varying studies of overall plume trajectory.

Acknowledgments

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Appendix

Extraction, Chromatographic, and ... Analyses

The sealed 2×10^{-5} kg/m^3 VOA ... soil samples and pretared solutions ... sieve shaker for 900s in the labor... down the soil structure and dissolv... into the methylene chloride. Abo... methylene chloride phase was th... VOA bottle by syringe and injec... Pasteur pipette filled with reagent ... strip dissolved water and particula... solution, which passed into $2 \times$ ... were sealed with Teflon-faced silic... caps and refrigerated at 4 C until t... chromatograph.

A volume of 10^{-9} m^3 of the w... methylene chloride solution was wi... ple vial and injected into a Varian 3... through a split/splitless injector usi... Gastight syringe equipped with a 26... and a plunger guide. A hot needle... featuring about 3×10^{-9} m^3 of p... exposure in the injector before syri... chromatograph was equipped w... HP-5 25m capillary column of 3.2 x... The injector temperature was 32...

-is provided concentrations of individ- :ne constituents. as discussed in the model studies of the vertical distribu- ate-phase components are contem- iverse City data base as well. in an the impact of solubility and vapor ristics on the transport of LNAPL ary fringe (Baehr 1987)

Conclusions

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:nd sleeve soil subsamples were sub- oride extraction and gas chroma- ielding surprisingly precise esti- cally integrated gasoline mass and the ination location in the capillary fringe precisions of 12 percent and 14 percent the two sampling locations and, in the locus of peak separate-phase pollution with the elevation where all pores mean pore size were saturated. This entration was about 0.2m thick at both the sleeve resolution of 0.03m was ve its vertical structure. The 0.1m bar- iy have obscured some details of the . so that replicate boreholes or half- warranted for vertical transport model- e of companion sleeve data. A single trusion with pint-size jars was judged pth-integrated, horizontally varying l plume trajectory.

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Appendix

Extraction, Chromatographic, and Gravimetric Analyses

The sealed 2×10^{-5} kg/m^3 VOA bottles containing the soil samples and pretared solutions were shaken in a Tyler sieve shaker for 900s in the laboratory to further break down the soil structure and dissolve the aviation gasoline into the methylene chloride. About 2×10^{-6} m^3 of the methylene chloride phase was then removed from the VOA bottle by syringe and injected into a 0.15m long Pasteur pipette filled with reagent grade sodium sulfate to strip dissolved water and particulates out of the organic solution, which [illegible] into 2×10^{-6} m^3 vials. The vials were sealed with [illegible]-faced silicone closures and screw caps and refrigerated at 4 C until their analysis in the gas chromatograph.

A volume of 10^{-9} m^3 of the water-free, gasoline-rich, methylene chloride solution was withdrawn from the sample vial and injected into a Varian 3500 gas chromatograph through a split/splitless injector using a 10^{-8} m^3 Hamilton Gastight syringe equipped with a 26-gauge bevel tip needle and a plunger guide. A hot needle injection was adopted, featuring about 3×10^{-9} m^3 of preceding air and a 5s exposure in the injector before syringe activation. The gas chromatograph was equipped with a Hewlett Packard HP-5 25m capillary column of 3.2×10^{-4} m I.D. fused silica. The injector temperature was 325 C and a split ratio of about 1:80 was employed with zero-grade nitrogen serving as the carrier gas at a rate of 3.3×10^{-8} m^3/s. The oven initial temperature of 33 C was held for 180 sec and then increased at a rate of 0.167 C/s for about 600s, sufficient for the arrival of the slowest aviation gasoline constituent. A flame ionization detector at 300 C sensed the separated constituents using an attenuation and range of 32 and 12. The results were tabulated on a Spectra Physics Chromjet integrator, then stored on an NEC Power Mate personal computer.

A laboratory standard consisting of 10 primary aviation gasoline constituents previously identified by the U.S. Environmental Protection Agency was prepared in accordance with the composition cited in Table 9. The 10 compounds accounted for more than 85 percent of the weathered aviation gasoline mass at the site, with retention times of less than 180s and saturated vapor densities (ρ_{SAT}) ranging from 0.05 to 0.65 kg/m^3 in magnitude. The identification of the compounds by their elution order was confirmed by GC/MS analysis of pure standards and comparison of electron impact mass spectra. The Environmental Engineering Hewlett Packard gas chromatograph (5890) mass spectrometer (5988A) was used to this end.

A range (0.05 percent to 3 percent) of standard solutions in methylene chloride was run to cover the variation of sample strengths encountered in the ana-